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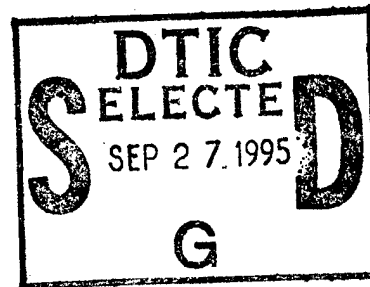
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Spectroscopy

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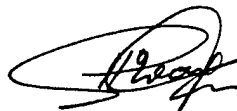
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# 1. Introduction

Development of a monitor to measure  $O_2$  and  $CO_2$  content in the expired air is the objective of this program. We use diode laser based combined wavelength and frequency modulation spectroscopy<sup>1</sup> (CWFMS) as the detection technique for this purpose. Both  $O_2$  and  $CO_2$  have near-infrared absorption band. For biomedical application, the accurate measurement of  $O_2$  concentration in the expired air is crucial for obtaining information about condition of the patient.

CWFMS is an advanced laser absorption spectroscopy and is the result of more than 14 years of research by a number of researchers. Unprecedented detection sensitivity has been achieved in a number of applications such as plasma diagnostic and trace gas detection. It combines the benefits of conventional wavelength modulation spectroscopy and the quantum noise limited frequency modulation spectroscopy. The use of diode lasers permits fabrication of small, rugged and long life instrument. Currently, room temperature diode lasers are available from 633 nm to 2.0  $\mu m$ . Near room temperature lasers are available upto 2.4  $\mu m$ .

The major challenge of this phase I program was to design a reliable portable expired air sensor based on CWFMS which can be used for detection of  $O_2$  and  $CO_2$ .  $O_2$  has an absorption band at 760 nm and  $CO_2$  has a relatively strong absorption band at 2.05  $\mu m$ . Both gases can be detected using room temperature diode lasers.

## ***1.1. Chemically Selective Sensitive Trace Gas Detection via Infrared Diode Laser Absorption Spectroscopy***

Most molecules possess an infra-red (IR) absorption spectrum which is sufficiently distinct to allow identification and discrimination among several species. Provided the transition is strong enough for each of the species of interest, IR absorption could thus form the basis for a versatile, sensitive, trace gas detection system. Table 1 lists the absorption bands of some important medical gases and their corresponding estimated detection sensitivity.

Gas	Absorption Band Center (cm <sup>-1</sup> /μm)	Sensitivity*
<sup>12</sup> CO <sub>2</sub>	4977.875/2.01	0.2 ppmv
<sup>13</sup> CO <sub>2</sub>	4887.391/2.05	20 ppmv
O <sub>2</sub>	13120.909/0.762	24 ppmv
NO <sup>+</sup>	3723.853/2.685	43 ppbv
H <sub>2</sub> O	7249.811/1.379	8.63 ppbv

Table 1. Detectable species and the corresponding absorption bands. \*Sensitivity are determined using 7 cm optical path, 1Hz detection bandwidth and a routinely achievable  $5 \times 10^{-7}$  absorbance limit.

## 1.2. Tunable Diode Lasers

Semiconductor diode laser technology makes possible the fabrication of single wavelength, tunable light sources, which can be designed to emit light at a specified wavelength. The tunability of all semiconductor lasers is based on the concept of band gap tuning in which the band gap energy and hence lasing frequency is adjusted by appropriate alloy composition of the compound semiconductor. These sources are well matched to sensitive detection requirements. They have very narrow band of emitted radiation, emit high enough laser intensity to allow quantum limited detection<sup>1-3</sup> and can be easily frequency and wavelength modulated through the laser injection current.<sup>4</sup> The latter feature is especially important when considering signal processing requirements.

The most versatile semiconductor technology for tunable laser sources is based on lead-salt semiconductors. These devices cover the 300 cm<sup>-1</sup> to nearly 3,000 cm<sup>-1</sup> (3.3-30μm) spectral range. However, this lasers required to be operated at cryogenic temperature and cause temperature controlling difficulties in the field. Using III-V compounds, room temperature diode lasers are available upto 2.0 μm. Near room temperature diode lasers are available upto 2.4 μm. These advanced lasers can be operated with thermoelectric coolers and simplifies the operation and permits fabrication of portable systems based on these lasers.

### 1.3. Modulated Diode Laser Spectroscopy

In order for diode laser absorption spectroscopy to be used in a sensitive chemical detection device, it is necessary to overcome some attendant difficulties in measuring the amount of light absorbed. Indeed the central difficulty is that even for relatively strong absorption lines,<sup>2</sup> little laser light will be absorbed and if one simply measures transmitted laser power through an absorbing gas, a host of noise sources will obscure the small reduction in transmitted light that the absorption causes. This limitation may be surmounted by employing a technique known as frequency modulation (fm) spectroscopy<sup>2,3</sup> wherein the laser frequency is modulated at rf frequencies. When the laser is tuned through a spectrally narrow feature, the fm light is converted to amplitude modulated light and the amplitude modulation may be measured with much higher sensitivity than the directly absorbed light. This approach allows the detection of absorbance as small as a few parts in  $10^8$ .<sup>1,3</sup>

Due to this high sensitivity, in almost all situations, false absorption features are detected as well.<sup>2,5-7</sup> These false features, especially transparent optical surface generated interference fringes, could be 2 to 3 orders of magnitudes higher than the theoretical limits.<sup>5-7</sup> This sensitivity limiting false features has been the practical sensitivity limiting factor for more than a decade.<sup>5</sup> Using another level of low frequency triangular wave modulation,<sup>5</sup> molecular absorption features can be separated from the false absorption features.<sup>7</sup> The overall modulation technique is especially well suited for use with diode lasers as their frequency may be modulated directly through the injection current. Figure 1 shows the basic principle of CWFMS. *The high sensitivity nature of the modulation technique can also be translated into high speed of operation.*

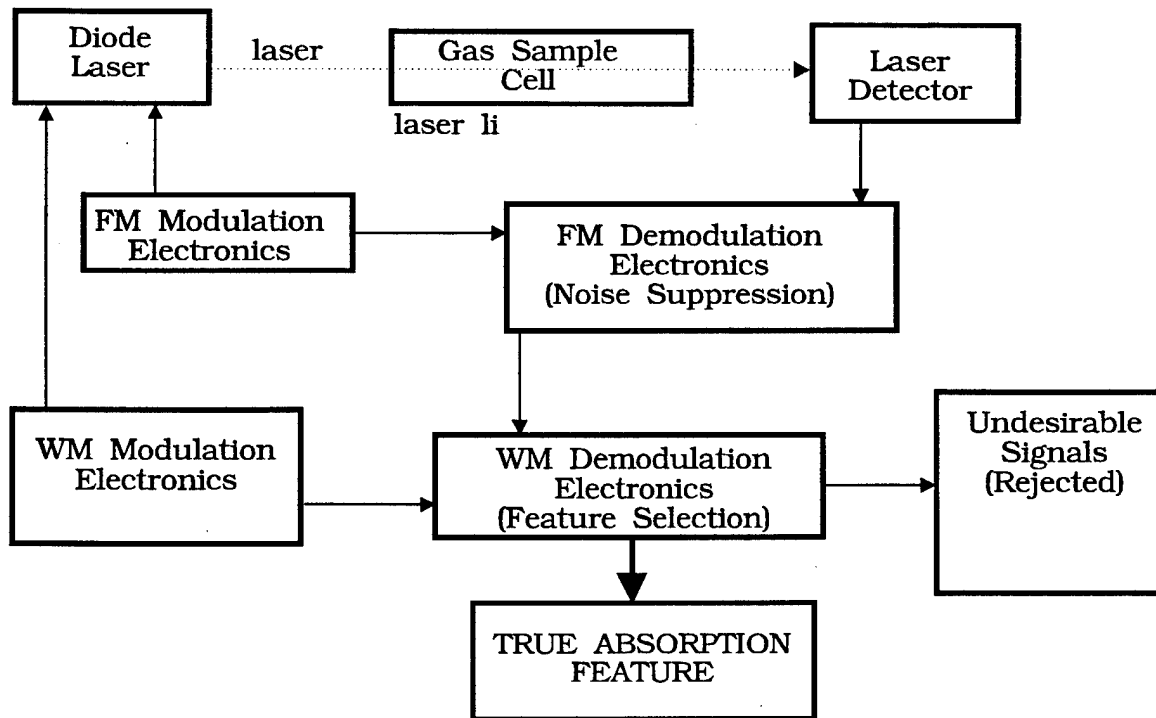


Figure 1. The basic principle of Combined Wavelength and Frequency Modulation Spectroscopy.

## 2. Proposed Work and Work Performed

### 2.1. Proposed Work

In phase I, we proposed to design and fabricate a prototype  $\text{CO}_2/\text{O}_2$  ratiometer and design a NO monitor. The work is undertaken in collaboration with Walter Reed Army Institute of Research (WRAIR).

### 2.2. Basic Detector Design

The detector design is based on CWFMS scheme. Figure 2 shows the simplified schematic of CWFMS. The major modules are the current source, the laser head, RF modulation source and the detector-demodulator module. In the design, the laser head is integrated to the gas cell. The Electronic Adder is integrated with the triangular wave generator and a custom made lock-in amplifier. The RF generator and the Bias Tee are integrated into one single circuit board.



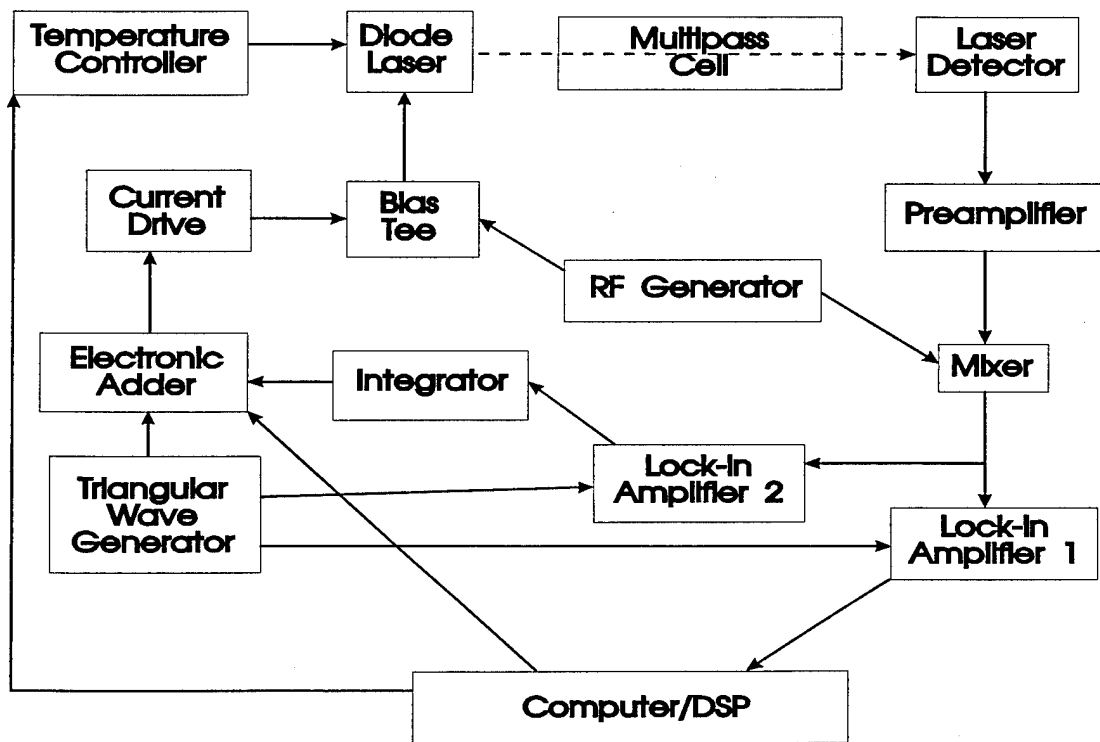


Figure 2. The schematic diagram of CWFMS.

## 2.3. First Generation Prototype

According to the communication with personnel (Dr. F. Pearce and Dr. W. Wiesmann) in WRAIR, high accuracy breath-by-breath  $O_2$  concentration measurement is the most important task to be completed. This requirement corresponds to a signal-to-noise ratio of at least 2000 to be achieved at 300 ms time-constant. Based on the schematic described in section 2.2, we fabricated a first generation  $O_2$  detector based on in-house fabricated modules and off-the-shelf lasers and components.

### 2.3.1. Individual Module Design and Fabrication

#### 2.3.1.1. Current Source

Diode lasers require stable current sources to operate. To implement CWFMS, the current source must be able to modulate the laser driving current in excess of 50 kHz. Currently, modularized

current source based on hybrid circuits are available commercially. Different diode lasers require current source capable of delivering current ranging from 50mA to 2A. We have acquired several modular current sources that fits our requirements and have full load current output of 100mA, 200mA and 1A respectively. A 3A bench top current source was available in PES laboratory. The testing results are listed in the Table 2.

Current Source	Power Supply Required	Total Power Consumption	Physical Space Required
100 mA	+5V	350mW to 500mW	1.5"x2.5"x3/4"
200 mA	+5V	600mW to 1W	1.5"x2.5"x3/4"
1A	+5V, +/-15V	3.1W to 7W	4"x5"x1"

*Table 2. Testing results for the current sources.*

#### **2.3.1.2. Temperature Controller**

The temperature controllers for thermoelectric coolers has been developed by us using hybrid circuits. The overall circuit integrates a 2A dual polarity current source and a close loop control circuit which utilizes a thermister as the temperature sensors. The space required is 2.5"x2.5"x1" and our test indicates a power consumption from 300 mW to 4W depends on the temperature control requirement of the particular diode used.

#### **2.3.1.3. The RF Modulation Source**

The module provides the necessary RF sources for modulating the laser as well as the local oscillator input of the demodulation module. PES has designed an RF source that provides a stable 7 dBm power for the local oscillator and also a power tunable RF output for modulating the laser diode. The tuning range is from -20dBm to 7dBm. The power requirement is up to 2.4W.

#### **2.3.1.4. The Detector-Demodulator Module**

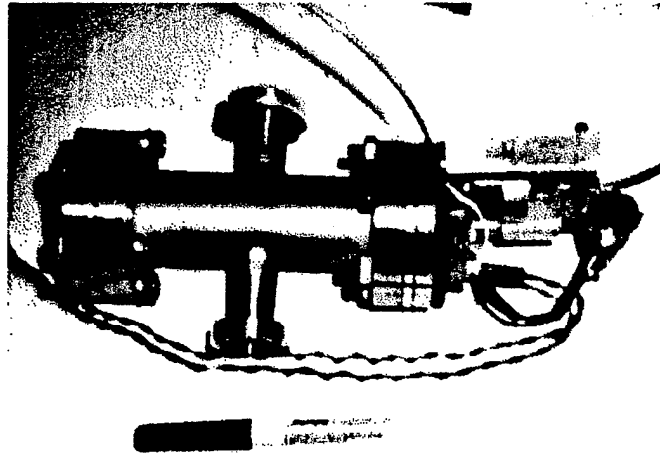
The performance of this module is very important to the detection sensitivity of the overall detection scheme. This module includes a high frequency photodetector, a high frequency trans-impedance amplifier, a high frequency mixer, low pass filter, a lock-in amplifier and a digital signal processor. The photodetector and the trans-impedance amplifier were integrated in a single

circuit board of 0.5"x1" and consumed a total of only 100mW. The photodetector and the trans-impedance amplifier were then integrated to the gas cell for minimum disturbance and maximum RF shielding.

The output of the trans-impedance amplifier is fed to the mixer and low pass filter which require a volume of 1"x1"x5/8". PES also has designed and fabricated a compact lock-in amplifier that has a bandwidth of up to 1Mhz. The power requirement for the lock-in amplifier is 1.5W. The size of the lock-in amplifier is 2.5"x3"x5/8". The digital signal processor is an off-the-shelf DSP board based on Texas Instrument TMS320C31 33 MFLOP (50 MFLOP enhancement available) signal processor which consumes only 1W of power. The DSP perform signal averaging and low frequency modulation-demodulation in addition to the CWFMS scheme. It also leaves room for high performance post spectrum acquisition signal enhancements once the enhancement algorithms are available.

#### **2.3.1.5. The Gas Cell**

The gas cell has a demanding set of mechanical and electrical specifications. We used low cost off-the-shelf ultra-high vacuum components for its development. The laser and detector mounts were tailor-made to suit this application. The cell integrates a 8 pass optical cell (10 pass maximum), the laser head and the photodetector for base performance. Commercially available cell costs as much as \$20k. Our current design cost about \$500. Figure 4 is a photograph of the first prototype of this cell. A marker is placed adjacent to the cell to indicate the size of the cell. The length of the cell is 5". In phase II, we will add a miniature pressure sensor and a miniature flow controller to the cell.



*Figure 3. The first prototype of the gas cell.*

### **2.3.2. Prototype Portable Gas Monitor**

All the modules were integrated into a new near infrared combined wavelength and frequency modulation spectrometer. Figure 4 shows the laboratory type setup of this spectrometer. The spectrometer was tested with atmospheric O<sub>2</sub> detection. The absorbance detection sensitivity achieved is  $2 \times 10^{-7}$ . A factor of 4 higher than the ultimate achieved in a laboratory unit. This sensitivity can be improved further by proper RF shielding. The spectrometer was tested for long term durability and stability for a period of 100 hours (approximately 4 days continuous running). Table 3 lists the testing results of these modules and some minor components.

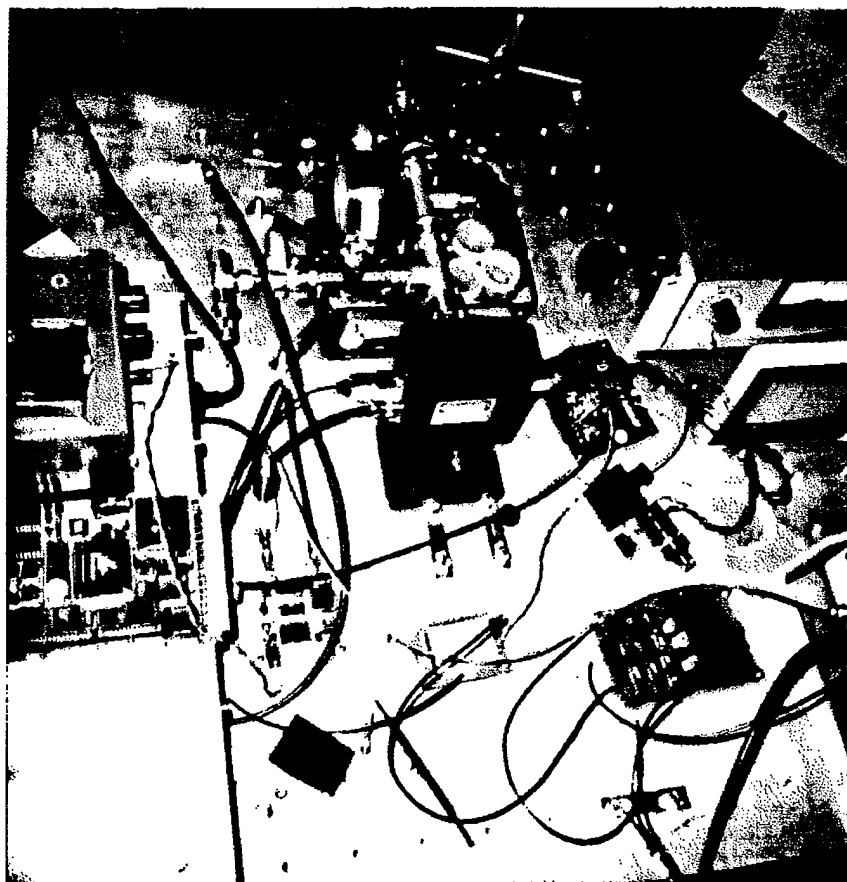
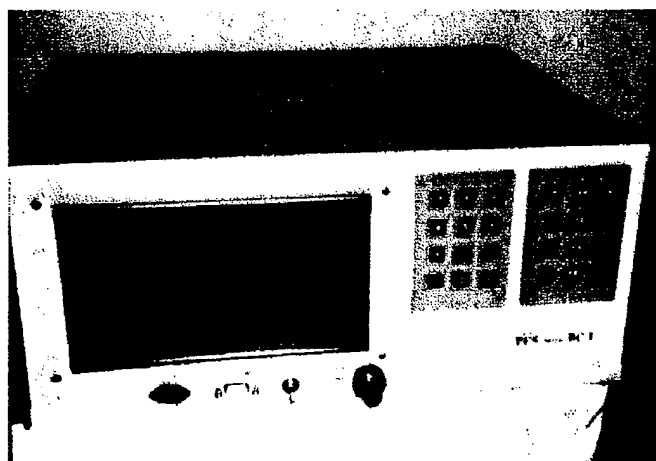


Figure 4. The laboratory type spectrometer set up for testing of the modules.

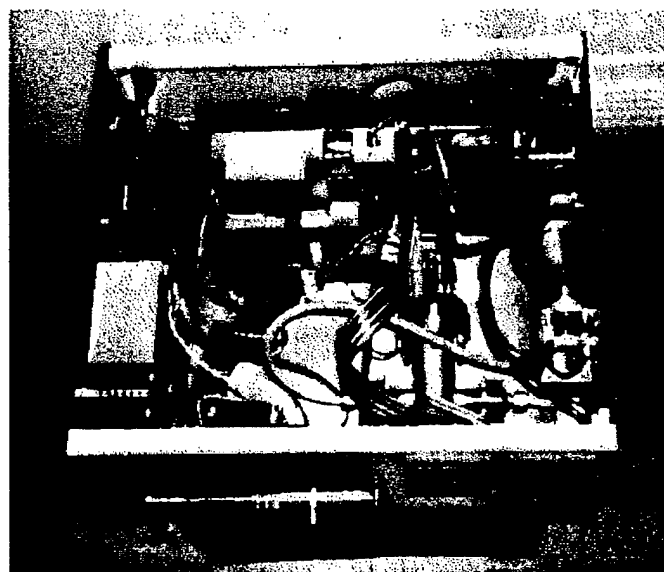
Module	Size	Power requirement	Component cost
Current source	<2"x3"x1"	<500 mW	\$180
Temperature controller	2.5"x3"x1.5"	<4W	\$150
Lock-in amplifier	3"x4"x0.5"	<1.92W	<\$150
RF modulation source	<3"x4"x0.5"	<2.55W	<\$560
Detector and preamplifier	<1"x1"x0.5"	<50mW	<\$250
Above Five Modules during 100 hours testing	N/A	<3.47W	N/A
Digital Signal Processor	<4.5"x7"x1"	<1W	<\$1300
User Interfacing CPU	credit card size	<210mW	<\$865
Display	various	<3W	<\$350

Table 3. Testing results of the various components of the CWFMS scheme for their functionality and power consumption.

After the modules were integrated and tested as a spectrometer, they were assembled into a bench top gas sensing unit. This unit was again tested with oxygen detection. This unit was designed to accommodate lasers from 700 nm to 2.2  $\mu\text{m}$  while the photodetector is capable of detecting laser light from 700 nm to 1.8  $\mu\text{m}$ . In this first design, the unit can detect  $\text{O}_2$ , CO,  $\text{CO}_2$ , and  $\text{NO}_2$  without modification. Figure 5 shows this prototype as well as its interior configuration. The size is 17"x14"x7". It is estimated that only about 60% of the volume is filled with components, which means with proper layout the volume of the unit can be reduced considerably.



*Figure 5a. The exterior view of prototype gas sensor.*



*Figure 5b. The interior view of the prototype gas sensor.*

### **2.3.3. Identified Practical Problems**

The prototype gas sensor was tested in-house and sent to WRAIR for testing. This system deliver signal-to-noise ratio of at least 5,000 at 1 Hz detection bandwidth when the laser was operated in sweeping mode. Acquisition time is however much longer than 300 ms. Long term testing results indicated improvements in the stability of the system are needed as well as the long term laser drifting effect must be reduced. We believed they are due to the temperature sensitivity of the hybrid circuits used in the system. As all the modules are placed inside a box, heat dissipation is not as good as in an open system. Also, a factor of 5 in sensitivity is lost due to the improper match of the off-the-shelf laser and the absorption line.

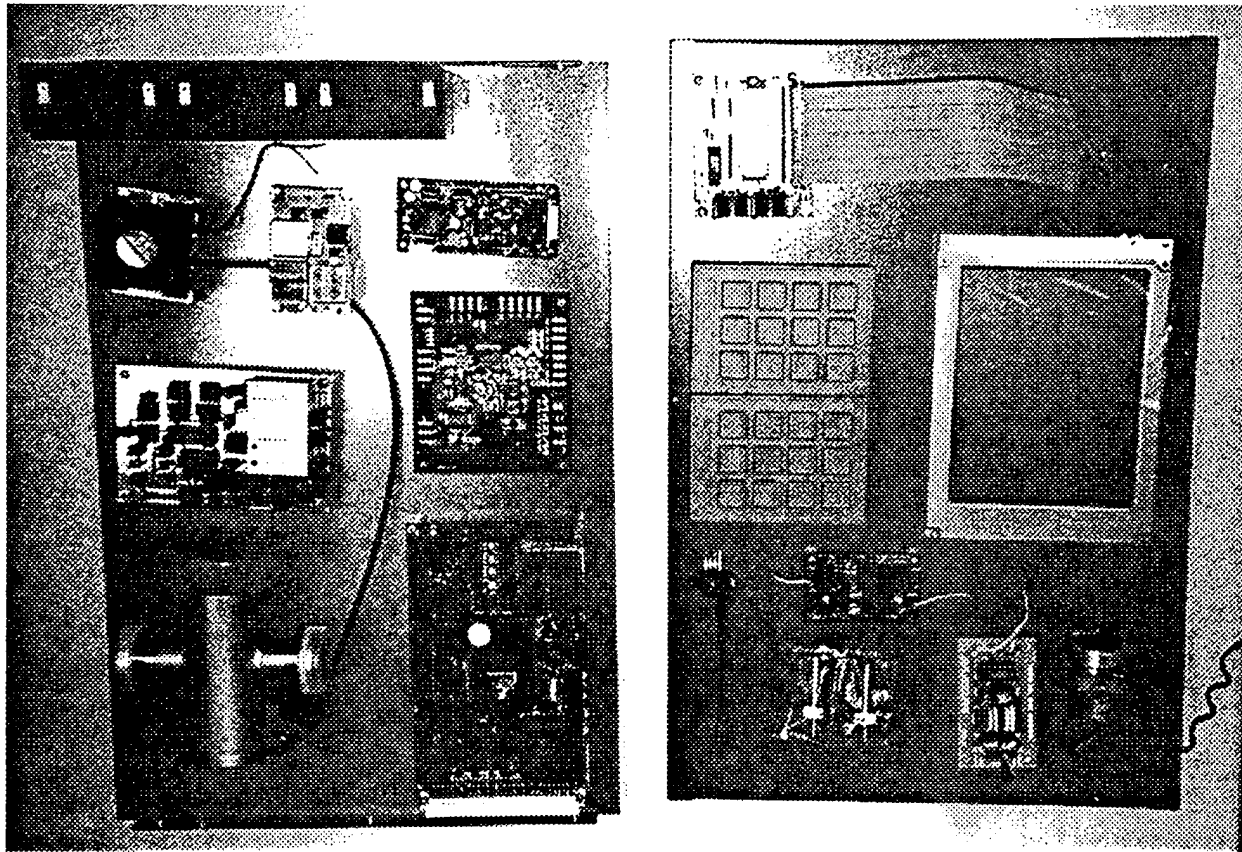
### **2.4. Second Generation Prototype**

Using the testing results of the first prototype, improvements in virtually all modules are on the way. First, the new prototype has a size of 5"x11"x14" which is only 46% in size of the first prototype. The improvements on the individual modules are the following:

- The current drive was integrated with the lock-in amplifier module and consumes a total power of 1.6 W. Power improvement is 33%. Size improvement is 60%.
- The lock-in amplifier circuit was redesigned to perform two orders lock-in amplification which is needed for laser line locking.
- The rf source was redesign with a power consumption of only 20% of the original design and has required only 25% of the original space.
- A stabilization miniature cooling fan was added to the temperature controller.
- The detector-amplifier modules was redesigned to include the measurement of laser power to compensate system fluctuation.
- The gas cell was modified to enhance mechanical stability.
- A miniature pressure sensor is added to compensate pressure fluctuation.
- Two miniature flow valves are added to control the air flow through the gas cell.

- Power supplies are consolidated and battery operation is implemented.

The initial testing results of these modules offer much higher stability as compare to the modules installed in the first prototype. The second prototype is currently being assembled. Figure 6 shows the collection of the major components and major modules. Figure 7 shows the new integrated lock-in amplifier. Figure 8 shows the first and the second RF source designs.



*Figure 6. Collection of the major modules and components of the second generation oxygen sensor prototype.*



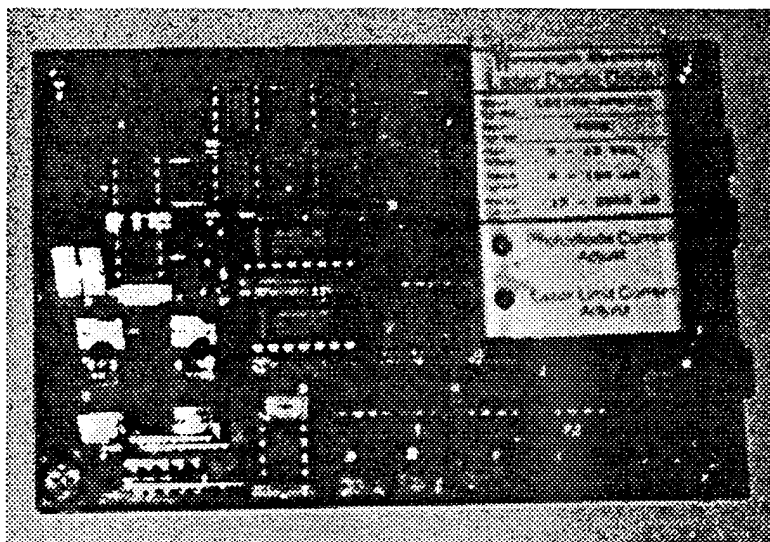


Figure 7. The new integrated lock-in amplifier. This prototype integrates two lock-in amplifier and the diode laser drive in a single board. Size improvement as compared to be first prototype is 60%.

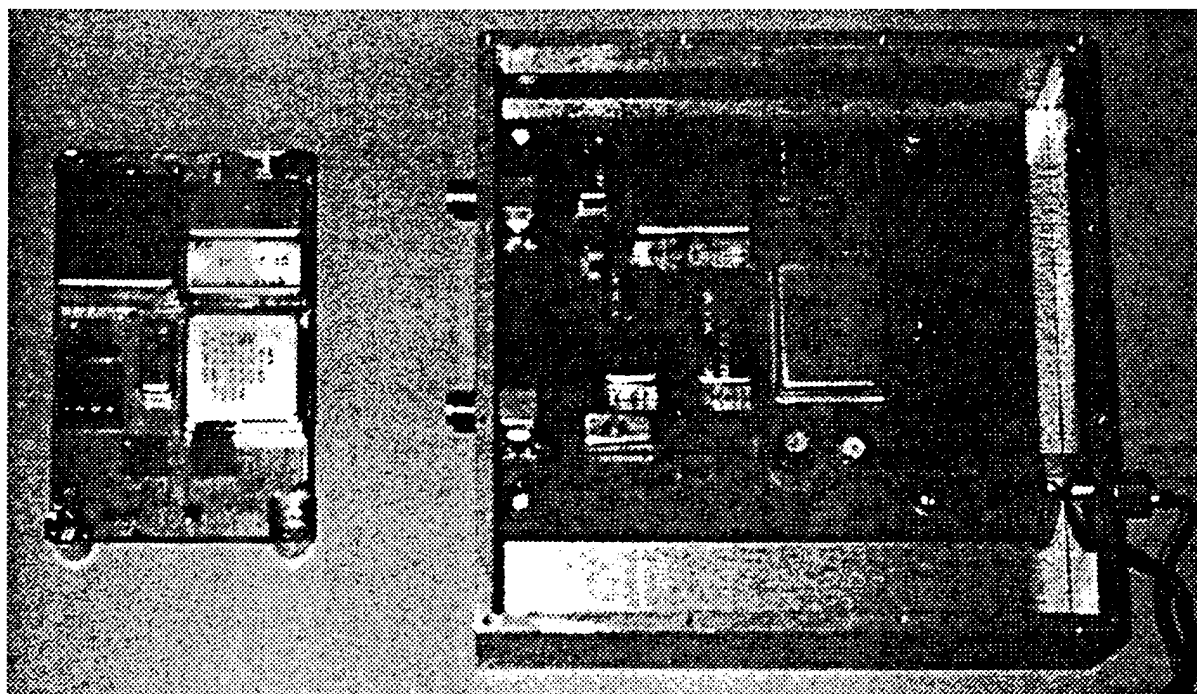


Figure 8. The first generation and the second generation rf-source module for the oxygen sensor. The second generation prototype is put on the left.

### 3. Conclusion

In this program, in the current stage, we completed the following:

- Designed and fabricated a custom made multipass cell.
- Tested individual modules.
- Fabricated the first prototype portable O<sub>2</sub> monitor.
- Tested the first prototype.
- First prototype has a size of 7x14"x17", 18-38 W power consumption.
- Identified practical problems for the first prototype and improve the design.
- Improved performance of individual modules.
- Added components for compensation of system instability.
- Power consumption as low as 10W (estimation on the second prototype).

The testing results of the first prototype indicate that the requirement for breath-by-breath monitoring of oxygen concentration can be achieved with the implementation of laser line locking technique. However, the stability problem of the laser controlling system must be resolved.

In the next few months, we will complete the second prototype for testing. The laser line locking technique invented<sup>1</sup> with CWFMS will be implemented. The miniature version of the oxygen sensor as well as the NO sensor will be designed.

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